

02-1-858



PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant: Raukas et al.

Serial No.: 10/665,257

Art Unit: 1755

Filed: 9/20/2003

Examiner: C. Koslow

For: Europium-Activated Barium Magnesium Aluminate Phosphor

Hon. Commissioner of Patents and Trademarks
Washington, D.C. 20231

Sir:

CERTIFICATE OF MAILING UNDER 37 CFR 1.8

I hereby certify that this correspondence is being deposited with the United States Postal Service as first class mail in an envelope addressed to: Commissioner for Patents, P.O. Box 1450, Alexandria VA 22313-1450.

October 5, 2005
Robert F. Clark

Robert F. Clark Reg. No. 33,853

DECLARATION UNDER 37 C.F.R. 1.131

This Declaration is being filed to establish completion of the invention in the above-identified application in the United States at a date prior to March 27, 2003, the effective date of WO 03/025089 and prior to March 19, 2003, the effective date of JP 2003-082344.

This declaration is being submitted prior to a final rejection.

We, as the below-named inventors, hereby declare:

1. That the persons making this declaration are inventors of the invention claimed in the above-identified application.
2. That the following exhibits establish a reduction to practice of invention in the United States prior to March 19, 2003 the earliest effective date of the references cited above.

Exhibit A is a copy of pages from our invention disclosure which demonstrate that the inventors had doped a europium-activated barium magnesium aluminate phosphor with silicon, hafnium and zirconium. The pages in the lower right-hand corner were witnessed and initialed by five persons on 9/27/02.

Exhibit B is a copy of laboratory notebook pages authored by Robert T. McSweeney, a joint inventor named below, which demonstrate a europium-activated barium magnesium aluminate phosphor had been doped with a range of Si, Hf, and Zr concentrations from 0.02 to 0.5 mol %. The date of the signatures in the lower right-hand corner of the pages indicates that these compositions were made at least as early as 9/27/02.

3. That all of the acts in the above exhibits occurred in the United States.

4. That, from the exhibits, it can be seen that the invention claimed in the above-identified application was made in the United States at least by September 27, 2002 which is prior to the effective dates of WO 03/025089 and JP 2003-082344 as given above.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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Inventor's name: Robert T. McSweeney Citizenship: USA
Signature: Robert T. McSweeney Date: 9/15/2005
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EXHIBIT A

Modification of BaMgAl₁₀O₁₇:Eu²⁺ (BAM) phosphor for higher radiation resistance in VUV/UV discharges by co-doping with tetravalent ions

1. Introduction and background

Blue-emitting BaMgAl₁₀O₁₇:Eu²⁺ (BAM) is widely used as a part of phosphor blends in most fluorescent lamps intended for white light generation. BAM also serves as a blue component in Plasma Display Panels (PDPs). Equally known are the shortcomings in its brightness and maintenance. In applications involving high VUV/UV fluxes, BAM emission is reduced at significantly faster rate over time than that of the other color components in the blend mixes or pixels. Therefore, a loss of lumens and a color shift in overall light output follow. This complicates manufacturing of products with satisfactory maintenance.

Theoretical and experimental investigations of various BAM compositions over the past few years as well as most recent tests of radiation and oxidation resistance of this and compounds belonging to the same hexa-aluminate family have yielded clues about the degradation mechanisms involved in phosphor maintenance. This Disclosure focuses on the improvement of the maintenance behavior of this phosphor under excitation by VUV light (particularly at 193 nm) that is caused by radiation induced defects. We believe that the maintenance of this phosphor can be improved by controlling (to an extent) the defect formation in the material. This fact has a beneficial influence on the long-term behavior of the phosphor in lamps of high 185/254 nm radiation intensity ratio and in Xe-discharge lamps or displays using 172 nm Xe₂⁺ band emission.

It has become evident through application tests and experiments of Mössbauer and optical spectroscopy that a prolonged exposure of BAM to radiation with photon energies above 5 eV (shorter wavelengths than 254 nm) causes reduction in brightness and changes the spectral power distribution of the phosphor output. These changes can be observed by spectroscopic methods after 500 hours of lamp exposure or can be simulated by a short period of high-intensity laser irradiation. We have used 193 nm excimer laser as the closest available high intensity source to the 185 nm mercury line in the fluorescent lamps. In addition to about 25% drop in phosphor brightness (upon 500-hour operation time), there is an increase in the long wavelength side of the emission band of the phosphor. This increase is not only relative (i.e. evident after normalization of the peak values before and after irradiation) but is absolute for wavelengths longer than ~550 nm. Both optical and Mössbauer spectroscopy measurements indicate a moderate conversion of Eu²⁺ into Eu³⁺. Emission from Eu³⁺ in this material is largely quenched. At this point, the cause for the increased emission intensity at long wavelengths is not clear and may be related to the defect formation in the host lattice itself. Additional evidence shows absorption and emission bands that are sensitive to annealing of the phosphor in oxygen-containing or neutral atmospheres. For example, the long wavelength emission tail can be removed during a short (about 1 hour) heating at moderate temperatures (e.g. 400 degrees C) in air. Similarly, there is a weak absorption

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in 400-500 nm spectral range in the virgin samples that can be reduced by exposing the powders to short-term heat treatment at 400 C in air. It is very likely that such additional absorption and emission bands are linked to electron and hole centers formed under reducing atmosphere during phosphor production and under ion/irradiation bombardment during lamp operation. For this purpose, oxygen and cation vacancies in the lattice can be named that further participate in capturing and releasing the electrons. Since the material recovers its brightness when annealed in a moderately oxidizing atmosphere we believe that oxygen vacancies in one or the other form play an important role in the degradation process.

2. Modification of chemical composition of BAM

We have tested the influence of incorporation of tetravalent ions to the radiation resistance of the in BAM phosphor. Apart from oxygen, BAM consists of cations Ba²⁺ (Eu²⁺), Mg²⁺ and Al³⁺. Replacing some of these original constituents by tetravalent cations supposedly reduces the probability of formation of oxygen vacancies. The creation of electron centers (oxygen vacancies that have captured zero, one or two electrons) can thus be reduced. These electron centers could compete with europium ions for 245 nm photons or could absorb visible radiation. In either case, they would affect the overall efficiency of BAM with time. Additionally, the oxygen vacancies with zero or one electron could capture electrons produced, for example, during the photoionization of Eu²⁺ to Eu³⁺ upon irradiation by 185 nm radiation. If the number of defects capable of capturing an electron from europium is comparable to the number of europium ions in the lattice, or becomes such in the course of irradiation, a serious reduction of the emission intensity of the material will follow over time.

BaMgAl₁₀O₁₇ samples of otherwise very tight stoichiometry were prepared with excess Hf⁴⁺, Zr⁴⁺ and Si⁴⁺ ions in the composition. The choice of these co-dopants was dictated by their stable 4+-valence state in varying conditions.

BaCO₃, BaF₂, Eu₂O₃, MgO, Al(OH)₃, HfOCl₂ 8H₂O, ZrO(NO₃)₂ and SiO₂ were used as precursor materials. BaF₂ added as a flux, substituting at the 10 molar % of the BaCO₃ input. Three different concentration levels of for all dopants were used: 0.02, 0.1 and 0.5 molar %, substituting in place of an equal molar portion of the Al(OH)₃, to the mentioned starting materials taken in stoichiometric ratio. The components were mixed together and additionally wet milled for 4 hours using YTZ beads, then filter dried, oven dried, crushed and fired. Prior to filtering the milled slurry was adjusted to have a basic pH, causing the soluble additives, primarily Hf or Zr precursors to precipitate in the mix and thus be retained on filtering. Synthesis of the samples was carried out at 1625°C in a production furnace with 75% H₂ / 25% N₂ atmosphere.

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3. Experimental evaluation and test results

Initial spectroscopic evaluation did not reveal any significant changes in the spectral output of the experimental samples when compared to the control. The brightness of all the samples stayed within a few percent of the standard BAM brightness that was used as a reference point. We then decided to test the highest concentration (0.5% at.) Hf, Zr and Si co-doped phosphor samples under 193 nm irradiation from Lambda-Physik Compex 110 excimer laser. The powders were hard-pressed into a recess in a copper sample holder that was placed in thermal contact with a cold finger of a CTI cryostat. No refrigeration of the sample was used during the irradiation in vacuum. Incident power density was kept constant at around 1.75 W/cm^2 . After a few trials, a ten-minute irradiation time was chosen to avoid nearly complete saturation in degradation of the top surface of the plaque upon prolonged exposures (e.g. an hour or more). In some cases, the procedure was repeated at least twice to assure the validity of the results. Compared to the control phosphor in the same conditions, the co-doped samples exhibited about 10% better brightness maintenance (measured as integrated visible radiance in the range of 350-600 nm under 250 nm excitation). These results are summarized in Table 1 (in relative units).

Table 1. Relative brightness results upon 250 nm excitation for as-received and irradiated samples.

Sample	Initial brightness	After irradiation	Difference to control
Control (undoped)	1.0	0.648	1.0
+0.5% Hf	1.0	0.707	1.091
+0.5% Zr	1.0	0.717	1.107
+0.5% Si	1.0	0.698	1.078

4. Conclusions

We believe that these preliminary results indicate that co-doping with tetravalent ions improves the VUV/UV radiation resistance of the BAM phosphor. Lamp testing of these phosphor samples is planned. It is likely that other tetravalent ions or ions with higher valence (5+, 6+ etc) may produce similar effects. The dopant concentrations and synthesis conditions need to be optimized.

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Project Number: 122014

September 30th, 2002

Monthly Progress Report for Improved BAM for PDP
R. McSweeney / T. Snyder

EXHIBIT B

Highlights:

In August we prepared, fired and processed nine BAM-compositions that were lightly doped with three levels of precursors designed to determine whether Hf⁺⁴, Zr⁺⁴ or Si⁺⁴ ions can enter the BAM lattice at some level and stabilize it against degradation under VUV radiation or against the effects of high temperature air annealing. Madis Raukis and Kalish Mishra at Cherry Hill requested that we prepare these compositions for testing with the expectation that if enough of any of these ions were soluble in the BAM lattice they would enter with associated oxygen and restrict the ability of the Eu⁺² ion to oxidize to Eu⁺³. Initial testing using a 193-excimer laser to damage these phosphors has demonstrated that the added plus four ions protect the phosphor to some extent, at least at the highest doping level.

Detailed Report:

MR-BAM:

Madis Raukis and Kalish Mishra at Cherry Hill requested that we prepare Hf⁺⁴, Zr⁺⁴ or Si⁺⁴ modified BAM compositions for testing with the expectation that if enough of any of these ions were soluble in the BAM lattice they would enter with associated oxygen and restrict the ability of the Eu⁺² ion to oxidize to Eu⁺³. In August we prepared, fired and processed nine BAM-compositions that were lightly doped with three levels of each of these precursors. These compositions were designed to determine whether Hf⁺⁴, Zr⁺⁴ or Si⁺⁴ ions can enter the BAM lattice at some level and stabilize it against degradation under VUV radiation or against the effects of high temperature air annealing.

All were prepared with the plus-four dopant substituting for the Al(OH)₃ input but since they were added at such low levels the weighing errors make these mixes essentially stoichiometric, forming (Ba_{0.90}Eu_{0.10})MgAl₁₀O₁₇, assuming that our assays of the input ingredients are correct. These blends, both in molar input and weight input, are shown below in Table I. Assays were determined by heating small amount of for the various input ingredients at temperatures between 800°C to 1500° for 1 to 2 hours to determine their respective weight losses. Assays for the BaCO₃ and BaF₂ lots were determined by heating to 850°C for 1 hour. Assays for the MgO and Eu₂O₃ lots were determined by heating 1000°C for 1.5 hours, and the Al(OH)₃, HfOCl₂·H₂O, Zr)(NO₃)₂ assays were determined by heating samples of each to 1500°C for 2 hours. The SiO₂ lot was assumed to have an assay of 1.000. The BaF₂ input was added as a flux, substituting for 10 molar percent of the BaCO₃ input. All the fluorine from the BaF₂ is given off during firing.

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Table I. MR-BAM Compositions with Hf, Zr and Si Precursors Added

Chemical:	BaCO ₃	BaF ₂	Eu ₂ O ₃	MgO	HfOCl ₂ ·8H ₂ O	ZrO(NO ₃) ₂	SiO ₂	Al(OH) ₃
Part No.	18900	1210	1511	18998	None	None	None	1021
Type	Prod.	Prod.	Prod.	standard	None	None	None	Prod.
Source	Solvay	Barco	RP	OSPI	Teled. WC	Ticon	Degussa	Malakoff
Lot #	322210	27485	010110	MgO13	SP97613A	5-AN-001	350	52014-AR
Assay	0.9965	0.9960	0.9856	0.9925	1.0050	0.8900	1.0000	0.9973
FW	197.339	175.327	351.920	40.304	409.517	231.233	60.0843	78.003
Molar Inputs								
Test	BaCO ₃	BaF ₂	Eu ₂ O ₃	MgO	HfOCl ₂ ·8H ₂ O	ZrO(NO ₃) ₂	SiO ₂	Al(OH) ₃
MR-BAM-1	0.800	0.100	0.050	1.000	0.0000	0.0000	0.0000	10.0000
MR-BAM-2	0.800	0.100	0.050	1.000	0.0002	0.0000	0.0000	9.9998
MR-BAM-3	0.800	0.100	0.050	1.000	0.0010	0.0000	0.0000	9.9990
MR-BAM-4	0.800	0.100	0.050	1.000	0.0050	0.0000	0.0000	9.9950
MR-BAM-5	0.800	0.100	0.050	1.000	0.0000	0.0002	0.0000	9.9998
MR-BAM-6	0.800	0.100	0.050	1.000	0.0000	0.0010	0.0000	9.9990
MR-BAM-7	0.800	0.100	0.050	1.000	0.0000	0.0050	0.0000	9.9950
MR-BAM-8	0.800	0.100	0.050	1.000	0.0000	0.0000	0.0002	9.9998
MR-BAM-9	0.800	0.100	0.050	1.000	0.0000	0.0000	0.0010	9.9990
MR-BAM-10	0.800	0.100	0.050	1.000	0.0000	0.0000	0.0050	9.9950
Weight Inputs								
Test	BaCO ₃	BaF ₂	Eu ₂ O ₃	MgO	HfOCl ₂ ·8H ₂ O	ZrO(NO ₃) ₂	SiO ₂	Al(OH) ₃
MR-BAM-1	39.61	4.40	4.46	10.15	0.020	0.000	0.000	195.53
MR-BAM-2	39.61	4.40	4.46	10.15	0.020	0.000	0.000	195.53
MR-BAM-3	39.61	4.40	4.46	10.15	0.102	0.000	0.000	195.52
MR-BAM-4	39.61	4.40	4.46	10.15	0.509	0.000	0.000	195.44
MR-BAM-5	39.61	4.40	4.46	10.15	0.000	0.013	0.000	195.53
MR-BAM-6	39.61	4.40	4.46	10.15	0.000	0.065	0.000	195.52
MR-BAM-7	39.61	4.40	4.46	10.15	0.000	0.325	0.000	195.44
MR-BAM-8	39.61	4.40	4.46	10.15	0.000	0.000	0.003	195.53
MR-BAM-9	39.61	4.40	4.46	10.15	0.000	0.000	0.015	195.52
MR-BAM-10	39.61	4.40	4.46	10.15	0.000	0.000	0.075	195.44

These ingredients were wet ball mill mixed in 1 L Nalgene bottles on a roller mill using approximately 1500 grams of 5 mm YTZ beads, 475 mls DI water, and the weight of input ingredients shown below in Table I. All were rolled for 4 hours using a speed setting corresponding to about 65 rpm or about 45 % of the Nc for the diameter of the bottles used. After milling the slurry was separated from the beads and the pH of the slurry was increased to above 8.00 by adding NH₄OH, if needed, to insure that any soluble additive, particularly the Hf and Zr precursors

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would precipitate out of solution. Surprisingly most of the slurries already had a high pH so the base addition was not needed. The slurries were then filter dried and oven dried at 120°C overnight. The dried cakes were then crushed in plastic bags and the powders added to high purity alumina boats.

These powders were fired in our new CM High Temperature Reduction furnace on 8/19/02 using a push rate corresponding to two moly carriers or four trays entering the furnace every 30 minutes, resulting in a six hour door-to-door time or about 1 hour at peak temperature. Three of the ten trays were broken but most of the powder from the broken trays was recovered. After firing the cakes were hand sieved past 35 mesh and added back to clean Nalgene mill bottles, along with about 1775 grams of 5 mm YTZ beads and 315 mls DI water. They were then milled for 1 hour, again at a speed setting corresponding to 65 rpm or 45% of the Nc for the bottle diameter. Following drying and sieving the samples were submitted for testing and X-Ray diffraction patterns were taken on all samples confirmed that the firing produced BAM or $(Ba_{0.90}Eu_{0.10})MgAl_{10}O_{17}$ with no minor phases visible in any of their XRD patterns. The particle sizes, BET surface areas, relative brightness and radiance levels are shown below in Table II. While some of the radiance levels are below 100% most of the reflectivity corrected radiances are very close to 100%. All were measured under 254 nm excitation. VUV measurements were done on the most highly doped samples and the control under Xenon excitation and all were at or above 100% of our SSX5 control. The OMA and VUV spectra for these tests are shown below in Figures 1 and 2.

Table II. Optical and Physical Properties of the MR-BAM Samples

NRF4 Type	Dopant Level	Radiance	Refl. Cr. Radiance	OMA Bghtns	X	Y	NS Mal.	SO Mal.	BET
							50%	50%	m ² /gm
BAM Std*	WOSX-654	100.0%	100.0%	100.0%	0.1489	0.0829	NM	NM	NM
MR-BAM-1M	none	94.2%	98.5%	81.6%	0.1514	0.0698	4.02	3.87	2.38
MR-BAM-2M	0.02% Hf	92.0%	97.2%	81.8%	0.1524	0.0719	3.87	3.77	2.90
MR-BAM-3M	0.10% Hf	96.2%	100.8%	82.5%	0.1522	0.0689	3.97	3.82	2.44
MR-BAM-4M	0.50% Hf	96.7%	98.7%	86.7%	0.1489	0.0724	4.20	4.06	2.09
MR-BAM-5M	0.02% Zr	98.7%	100.2%	83.7%	0.1511	0.0681	4.27	4.00	1.77
MR-BAM-6M	0.10% Zr	99.9%	100.9%	84.5%	0.1507	0.0679	4.45	4.08	1.66
MR-BAM-7M	0.50% Zr	100.0%	100.0%	86.7%	0.1513	0.0698	4.53	4.20	1.46
MR-BAM-8M	0.02% Si	99.8%	99.8%	81.6%	0.1506	0.0653	4.45	4.08	1.38
MR-BAM-9M	0.10% Si	100.2%	100.2%	80.0%	0.1502	0.0636	4.61	4.06	1.43
MR-BAM-10M	0.50% Si	99.9%	101.7%	81.0%	0.1506	0.0647	4.30	4.09	1.76

These samples are scheduled for VUV and heat degradation testing when time permits. Portions have been shipped to Madis Raukas at Cherry Hill for further testing and preliminary degradation testing using a 193 excimer laser show that the most heavily doped samples are about 10% better than the control after being subjected to a similar level of 193 excimer radiation.

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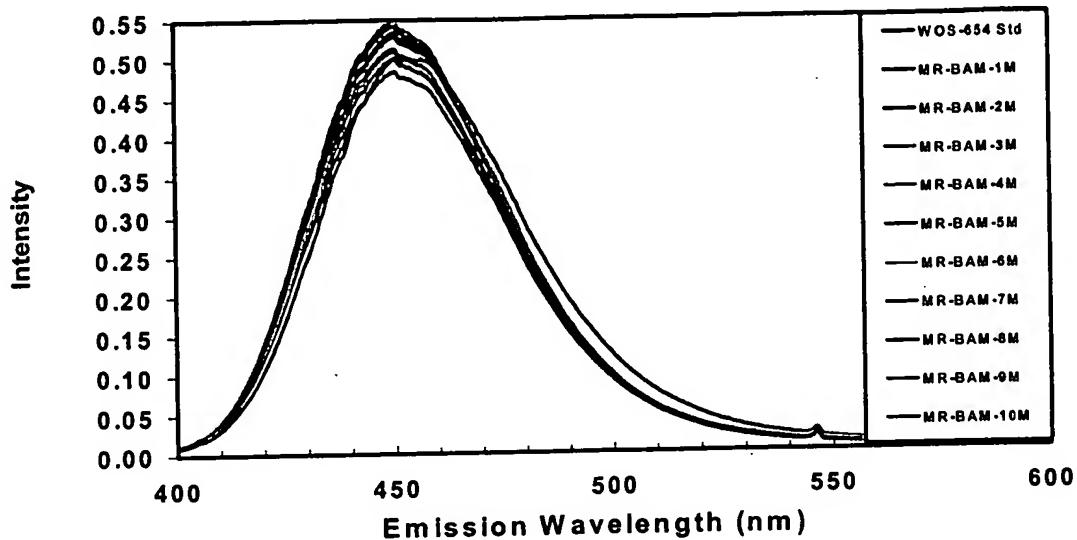


Figure 1. OMA Spectra for MR-BAM (10% Eu Doped) with Zr, Hf or Si

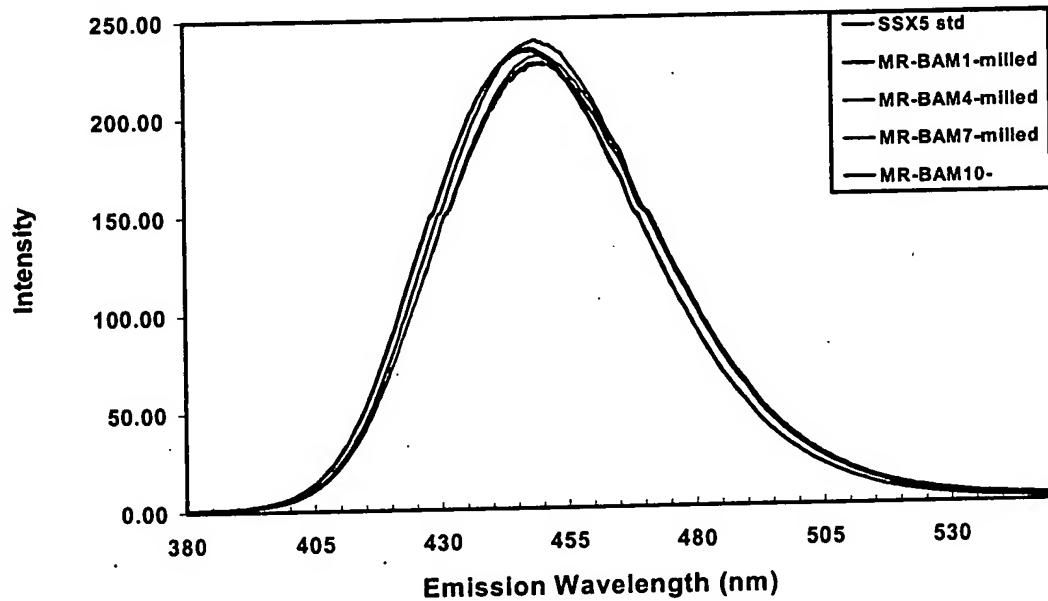


Figure 2. VUV Spectra for MR-BAM samples

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RELATED NOTEBOOKS & REF.

Batch numbers BAM with Hf, Zr, or Si substituting for Al series for Media Rautas and Kishore Mishra.

Chemical	BaCO ₃	BaF ₂	Ba ₂ O ₃	MgO	HfOCl ₄ ·8H ₂ O	ZrO(NO ₃) ₂	SiO ₂	Al(OH) ₃
Part No.	16900	1810	1511	15988	None	None	None	1021
Type	Production	Production	standard	None	None	None	Production	
Source	Sohva	Barco	OSPR	Teknodyn WG	Ticon	Dowcorning	Mitsubishi	
Lot #	222118	27451	210119	MgC013	EPF712A	JLH4-001	203	820144R
Alloy	0.9993	0.9990	0.9938	0.9923	1.0020	0.9900	1.0000	0.9873
FW	197.223	173.227	251.223	40.204	408.811	231.333	65.043	78.003

Test	BaCO ₃	BaF ₂	Ba ₂ O ₃	MgO	HfOCl ₄ ·8H ₂ O	ZrO(NO ₃) ₂	SiO ₂	Al(OH) ₃
MR-BAM-1	8.800	8.100	0.020	1.000	0.0000	0.0000	0.0000	10.0000
MR-BAM-2	8.800	8.100	0.020	1.000	0.0021	0.0000	0.0000	8.8986
MR-BAM-3	8.800	8.100	0.020	1.000	0.0019	0.0000	0.0000	8.8980
MR-BAM-4	8.800	8.100	0.020	1.000	0.0020	0.0000	0.0000	8.8982
MR-BAM-5	8.800	8.100	0.020	1.000	0.0000	0.0002	0.0000	8.8986
MR-BAM-6	8.800	8.100	0.020	1.000	0.0000	0.0010	0.0000	8.8986
MR-BAM-7	8.800	8.100	0.020	1.000	0.0000	0.0030	0.0000	8.8980
MR-BAM-8	8.800	8.100	0.020	1.000	0.0000	0.0000	0.0010	8.8980
MR-BAM-9	8.800	8.100	0.020	1.000	0.0000	0.0000	0.0030	8.8980
MR-BAM-10	8.800	8.100	0.020	1.000	0.0000	0.0030	0.0000	8.8980

BF =	Ba _{1-x} Eu _x Si _{1-y} Mg _{0.1} Al _{0.1} Fe _{0.7} Y ₂ O ₁₂ Si _{0.8}	Total
Compound	BaCO ₃	BaF ₂
Mole Ratio	0.8000	0.0500
Weight-gms	38.808	4.401
Weight Fraction	0.1558	0.0173
Compound Wt. Loss	0.2256	0.1300
Fractional Wt. Loss	0.0352	0.0023

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BF =	Ba _{1-x} Eu _x Si _{1-y} Mg _{0.1} Al _{0.1} Fe _{0.7} Y ₂ O ₁₂ Si _{0.8}	Total
Compound	BaCO ₃	BaF ₂
Mole Ratio	0.8000	0.0500
Weight-gms	38.808	4.401
Weight Fraction	0.1558	0.0173
Compound Wt. Loss	0.2256	0.1300
Fractional Wt. Loss	0.0351	0.0023

BF =	Ba _{1-x} Eu _x Si _{1-y} Mg _{0.1} Al _{0.1} Fe _{0.7} Y ₂ O ₁₂ Si _{0.8}	Total
Compound	BaCO ₃	BaF ₂
Mole Ratio	0.8000	0.0500
Weight-gms	38.808	4.401
Weight Fraction	0.1558	0.0173
Compound Wt. Loss	0.2256	0.1300
Fractional Wt. Loss	0.0352	0.0023

BF =	Ba _{1-x} Eu _x Si _{1-y} Mg _{0.1} Al _{0.1} Fe _{0.7} Y ₂ O ₁₂ Si _{0.8}	Total
Compound	BaCO ₃	BaF ₂
Mole Ratio	0.8000	0.0500
Weight-gms	38.808	4.401
Weight Fraction	0.1558	0.0173
Compound Wt. Loss	0.2256	0.1300
Fractional Wt. Loss	0.0352	0.0023

BF =	Ba _{1-x} Eu _x Si _{1-y} Mg _{0.1} Al _{0.1} Fe _{0.7} Y ₂ O ₁₂ Si _{0.8}	Total
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Wet Ball Mill Blending:

1. Add about 200-270 grams of each blend to a 1,000 ml PP bottle.
2. Add 475 grams DI water to bottle.

3. Shake up bottle to wet all of the powder.

4. Add 1,300 gms 10 mm YTZ beads.

5. Ball mill 4 hrs at 1,400 speed setting, Abbe M2, about 65 RPM.

6. Add stonex to 20 plastic sleeves into receiver pan to separate mill media.

7. Add stonex to 4 L plastic beaker and add enough ammonium hydroxide to raise the pH to 8.00.

8. Filter dry三星then oven dry.

9. Crush dry三星then oven dry.

10. Add powder to preweighed filter trays, weigh and oven dry overnight at 105°C.

11. Reweigh dried powders in trays after cooling to room temperature.

No Devan 621A added.

Recut - Med-BAM Test - Glance - 08/27/02

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G 34857 - 42

HF-S4:

08/19/02

Fired in new HT Reduction furnace, mostly on 2nd shift at set temperature of 1607°C. Finally reaches the 1625°C set temperature for the middle zones. Powders were not dried overnight in boats prior to firing. Just standard drying after filtering. No sieving just crushed in a bag and loaded into trays.

Powders were not dried overnight in boats prior to firing. Just standard drying time, no heating.												
08/19/02	Empty	Full	After	Unfired	After	Fired	Actual	Actual	Theo.	Diff.	Act-Theo	
HF-S4	Tray	Tray	Drying	Powder	Firing	Powder	Wt. Frac.	Wt. Frac.	Wt. Frac.	Act-Theo	Wt. Frac.	
Blend	Type	Weight	Tray	Weight	Weight	Weight	Left	Lost	Lost	Lost	Lost	
MR-BAM1	A-VMcD	389.25	635.82	246.57	635.82	246.57	557.57	168.32	0.6826	0.3174	0.3066	0.0108
MR-BAM2	A-VMcD	382.79	633.03	250.24	633.03	250.24	554.00	171.21	0.6842	0.3158	0.3066	0.0092
MR-BAM3	A-VMcD	374.53	626.44	251.91	626.44	251.91	626.44	251.91	1.0000	0.0000	0.3066	-0.3066
MR-BAM4	A-VMcD	389.00	641.09	252.09	641.09	252.09	561.85	172.85	0.6857	0.3143	0.3069	0.0074
MR-BAM5	A-VMcD	346.39	595.61	249.22	595.61	249.22	516.59	170.20	0.6829	0.3171	0.3066	0.0105
MR-BAM6	A-VMcD	351.81	602.02	250.21	602.02	250.21	523.10	171.29	0.6846	0.3154	0.3066	0.0088
MR-BAM7	A-VMcD	382.58	633.88	251.30	633.88	251.30	554.10	171.52	0.6825	0.3175	0.3068	0.0106
MR-BAM8	A-VMcD	404.06	653.21	249.15	653.21	249.15	575.20	171.14	0.6869	0.3131	0.3066	0.0065
MR-BAM9	A-VMcD	379.72	630.85	251.13	630.85	251.13	630.85	251.13	1.0000	0.0000	0.3065	-0.3065
MR-BAM10	A-VMcD	350.49	601.35	250.86	601.35	250.86	601.35	250.86	1.0000	0.0000	0.3065	-0.3065

Fired in high purity alumina trays from Vesuvius McDaniel previously used for BMBL series.

Average Excess Weight Loss:

Right main pusher setting under feed table set to 20.

Left pusher control set to 4 x 10 sec and right pusher control set to 10 X 10 seconds. 4 seconds of push per 14 seconds of cycle.

Pushes two carrier or four trays about every 30 minutes

Fusco holds two carrier of four trays about every 30 minutes.

Furnace holds 24 mud carriers with two trays each so these pusher settings result in a 3 hour 30 min. 40 sec. time.

Slow down with a break until all of your tasks are complete.

Milling: Post Firing Wet Ball Milling:

1. Hand sieve fired cakes past -35 mesh.
 2. Add sieved powder to 1 L Nalgene bottle, about 160 to 170 grams in each.
 3. Add 1775 grams of 5 MM YTZ balls to bottles.
 4. Add 300 to 315 mls DI water to bottles.
 5. Mill for 1 hour on Abbe Lab Mill, speed setting 1600. About 65 RPM.
 6. Filter dry and oven dry these samples overnight.

RMcS Madis-BAMTest Firing&Milling 09/27/02